

Union of Soviet  
Socialist  
Republics



Council of  
Ministers of the  
USSR, State  
Committee on  
Inventions and  
Discoveries

## Patent Specification for an inventor certificate

(11) **464602**

(61) Dependent on inventor certificate No. (blank)

(22) Filing Date: 03-Jan-1974

(21) Appl. No.: 1988192/23-5

(32) Priority:

Published: 25-March-1975, Bulletin No. 11  
Specification Published: 08-September-1975

(51) Int. Cl. C 08g 33/20

(53) Universal Decimal  
Classification: 678.84  
(088.8)

(72) Inventor(s): Korneev, N. N.; Lelyukhina, Yu. L.; Korabelnikova, E. G.; Chepkasova, A. S.

(71) Applicant: --

### (54) Title of the Invention: Method for Producing Polyalkyl Halogenoalumoxanes

The invention relates to a method for producing polyalkyl halogenoalumoxanes, which can be used as catalyst components during polymerization of polar monomers, diene hydrocarbons, and olefins.

There is a known method for producing a polyalkyl halogenoalumoxane by subjecting the corresponding halo-substituted aluminum alkyls to slow hydrolysis.

According to the method proposed herein, polyalumoxanes of the general formula



(where R is an alkyl, and n is 25 to 30) are reacted with an aluminum halide at a temperature of 0 to 150°C in a hydrocarbon solvent medium and an inert gas atmosphere in order to improve the yield and purity of polyalkyl halogenoalumoxanes.

The polyalkyl halogenoalumoxanes are obtained with a satisfactory yield after the solvent and aluminum dihalide have been removed at a reduced pressure. The reaction may also be conducted when the alkyl groups in the polyalkyl alumoxane are completely substituted by a halogen.

### Example 1

26 g of  $\text{AlCl}_3$  was introduced in an inert gas stream into the round-bottom flask (volume: 0.25 L) of a rotary evaporator equipped with a rotating stirrer, back-pressure funnel, and reflux cooler; and 60 mL of a 50% solution of polyisobutyl alumoxane in benzene was then metered out from the funnel. The reaction mass thus obtained was boiled under stirring for 1 hour at a temperature of  $80^\circ\text{C}$ . The solvent (benzene) and isobutyl aluminum dichloride were then distilled off at a temperature of  $150^\circ\text{C}$  and a residual pressure of 0.5 mm Hg.

The product was 12.5 g of chlorinated polyalumoxane, which as a yellowish powder soluble in benzene. The polymer yield was 83.3%.

Composition of the product obtained

Calculated, %: O 19.3, Al 33.7, Cl 47.0

Molecular weight: 2160

Actual, %: C 2.0, O 19.6, Al 33.4, Cl 46.0

Molecular weight: 2200

Chemical formula of the product



### Example 2

8.45 g of  $\text{AlCl}_3$  and 80 mL of a 30% solution of polyisobutyl alumoxane in benzene were introduced in the same manner as in example 1, a reaction was performed, and impurities were removed thereafter. This yielded 16.2 g of a product. The polymer yield was 75%.

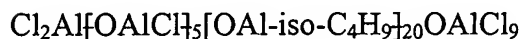
Composition of the product obtained

Calculated, %:  $\text{C}_4\text{H}_9$  43.8, O 16.05, Al 27.9, Cl 12.25

Molecular weight: 2600

Actual, %: C<sub>4</sub>H<sub>9</sub> 45.2, O 16.3, Al 26.5, Cl 12.0

#### Chemical Formula of Synthesized Polyalkyl Halogenoalumoxane



#### Example 3

30 g of polyethyl alumoxane was dissolved in 30 g benzene. The resulting solution was poured into 60 g of AlBr<sub>3</sub>, and the reaction mass was stirred and heated in the same manner as in example 1. The solvent was then removed at a temperature of 150°C and a reduced pressure. This yielded 40 g of a product with the following chemical formula.



The polymer yield was 97%.

#### Composition of polyalkyl halogenoalumoxane

Calculated, %: C<sub>2</sub>H<sub>5</sub> 16, O 15.4, Al 27, Br 41.6

Molecular weight: 2700

Actual, %: C<sub>2</sub>H<sub>5</sub> 15, O 15.7, Al 27.2, Br 42.1

#### Example 4

32.6 g of AlCl<sub>3</sub> was introduced into a four-neck flask that had a capacity of 0.5 L and was equipped with a stirrer, thermometer, dropping funnel, and a reflux cooler, and the system was cooled to -10°C. 150 mL of diethyl ether was slowly added from the dropping funnel so that the temperature did not exceed 0°C.

30 g of a 50% solution of polymethyl alumoxane in benzene was added at 0°C to the resulting aluminum chloride esterquat. All the solvents were dried before being used. The reaction mass was kept at 0°C for 30 minutes, the temperature was then raised to 35°C, and distillation of the solvents was started while the temperature was being raised to 100°C. The solvent was then distilled in a vacuum at 150°C and a residual pressure of 2 mm Hg in a rotary apparatus. 14 g of a solid white powder was obtained. The powder had a molecular weight of

1780 and contained 33.6% Al, 18.9% O, and 57.5% Cl. The product thus obtained dissolved well in benzene. The structures of all the compounds thus obtained were confirmed based on IR spectra.

### Claims

A method for producing polyalkyl halogenoalumoxanes, characterized in that polyalumoxanes of the general formula



(where R is an alkyl, and n is 25 to 30) are reacted with an aluminum halide at a temperature of 0 to 150°C in a hydrocarbon solvent medium and an inert gas atmosphere in order to improve the yield and purity of the polyalkyl halogenoalumoxanes.